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10/676,182

09/30/2003

Steven Verhaverbeke

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EXAMINER

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UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE BOARD OF PATENT APPEALS
AND INTERFERENCES

Ex parte STEVEN VERHAVERBEKE

Appeal 2008-3761
Application 10/676,182
Technology Center 1700

Decided: November 26, 2008

Before EDWARD C. KIMLIN, LINDA M. GAUDETTE, and
KAREN M. HASTINGS *Administrative Patent Judges*.

GAUDETTE, *Administrative Patent Judge*.

DECISION ON APPEAL

This is an appeal from the rejection of claims 1-5, 9-14, 17-20, 24-30, 34-38, 40, 42, 43, and 45.¹ We have jurisdiction under 35 U.S.C. § 6(b).

¹ All of the claims stand finally rejected with the exception of claims 36 and 37 which were subject to a new ground of rejection in the Examiner's Answer mailed July 11, 2007. *See infra* p. 4, ground of rejection 7. In response to the new ground of rejection, Appellant elected to maintain the appeal and filed a substitute Appeal Brief on September 10, 2007 (hereinafter "App. Br."). (*See* 37 C.F.R. § 41.39(b)(2)). The Examiner responded by remailing the Answer on November 5, 2007 (hereinafter "Ans.").

We AFFIRM.

The invention is directed to a method of removing a residue from an etched substrate surface in which “[t]he wash solution remains isolated from the cleaning solution.” (Spec. [0008].) The method is advantageous in that the concentration of the cleaning solution remains consistent from one substrate to another and contamination of substrates by particles removed from previously cleaned substrates is avoided. (Spec. [0007].) Claim 1 is illustrative of the invention and is reproduced below:

1. A method for removing a residue from a substrate surface, comprising:

 mixing an aqueous solution comprising sulfuric acid and hydrofluoric acid, wherein a concentration of the sulfuric acid in the aqueous solution is about 70% or less by weight, with a hydrogen peroxide solution to produce an intermediate solution at a predetermined temperature of about 3°C or less higher than temperatures of the aqueous solution and the hydrogen peroxide solution,

 diluting the intermediate solution with water to form a cleaning solution, wherein the cleaning solution comprises:

 hydrogen peroxide at a concentration within a range from about 1 % to about 15% by weight;

 sulfuric acid at a concentration within a range from about 1% to about 10% by weight; and

 hydrogen fluoride at a concentration within a range from about 10 ppm to about 1,000 ppm;

 applying an aliquot of the cleaning solution to a substrate surface for a time period; and

 rinsing the aliquot from the substrate surface with water to form a wash solution.

The Examiner relies on the following prior art references to show unpatentability:

Ramachandran	WO 02/10480	Feb. 7, 2002
Rath (“Rath ‘074”)	6,630,074	Oct. 7, 2003
Rath (“EP ‘081”) ²	EP 0 918 081 A1	Dec. 26, 1999
Kuhn-Kuhnenfeld	4,100,014	Jul. 11, 1978
Gotoh	5,650,041	Jul. 22, 1997
Oonishi	6,273,959	Aug. 14, 2001

Appellant requests review of the following grounds of rejection³:

1. claims 1, 2, 5, 9-12, 14, 20, and 24-27 under 35 U.S.C. § 103(a) as unpatentable over Ramachandran;
2. claims 1, 2, 5, 9-12, 14, 20, and 24-27 under 35 U.S.C. § 103(a) as unpatentable over Rath ‘074.
3. claims 1, 2, 5, 9-12, 14, 20, 24-27, and 38⁴ under 35 U.S.C. § 103(a) as unpatentable over EP ‘081.
4. claims 1, 2, 5, 9, 10, 14, 20, 24, 25, and 38 under 35 U.S.C. § 103(a) as unpatentable over Kuhn-Kuhnenfeld;

² EP ‘081 is the foreign equivalent of Rath ‘074. We refer to these references collectively as “Rath” and, for convenience, citations in the findings of fact are made only to Rath ‘074.

³ The following grounds of rejection have been withdrawn by the Examiner (Ans. 16-17): claim 43 under 35 U.S.C. § 103(a) as unpatentable over Rath ‘081; claim 37 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran or Kuhn-Kuhnenfeld in view of Gotoh; and claim 36 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran in view of Oonishi.

⁴ It is unclear to us why claim 38 was included in this ground of rejection, but was not rejected over Rath ‘074. *See supra* note 2.

5. claims 3, 4, 17-19, 29, 30, 34, 35, 40, 42, 43, and 45 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran or Kuhn-Kuhnenfeld in view of Gotoh;

6. claims 13 and 28 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran in view of Oonishi⁵; and

7. claims 36 and 37 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran in view of Gotoh as applied to claim 34, and further in view of Oonishi.⁶

Appellant traverses each of the first five grounds of rejection on the basis of the same limitations found in each of independent claims 1, 14, and 29. (App. Br. 10-21.) With respect to the sixth and seventh grounds of rejection, Appellant relies on the same arguments presented in connection with the independent claims. (App. Br. 22; Reply Br. 11-12.)

Appellant does not dispute the Examiner's finding that each of the primary references discloses a cleaning solution comprising hydrogen peroxide at a concentration within a range from about 1% to about 15% by weight; sulfuric acid at a concentration within a range from about 1% to about 10% by weight; and hydrogen fluoride at a concentration within a range from about 10 ppm to about 1,000 ppm. Nor does Appellant dispute

⁵ Appellant does not expressly request review of, or present arguments in response to this ground of rejection in the Appeal Brief (*see* App. Br. 9) as required under 37 C.F.R. § 41.37(c)(1)(vii). However, this appears to have been an inadvertent error. Appellant does include claims 13 and 28 in the list of appealed claims. (App. Br. 5.) In addition, Appellant expressly requested review of this ground of rejection in the Reply Brief (pp. 3 and 10-11) and in the original Appeal Brief, entered on March 2, 2007.

⁶ The Examiner identifies this rejection as a new ground of rejection (Ans. 15-16). *See supra* note 1.

the Examiner's finding that the primary references disclose applying the cleaning solutions to a substrate in the manner recited in each of independent claims 1, 14, and 29. Rather, Appellant argues that none of the references, alone or in combination, disclose or suggest preparing a cleaning solution by first forming an intermediate solution in the manner claimed (i.e., in accordance with the step(s) recited prior to the "diluting" step).⁷

The issue presented for our review is: has Appellant shown reversible error in the Examiner's conclusion that it would have been obvious to one of ordinary skill in the art at the time of the invention to have manipulated the conditions (e.g., concentrations and rates of addition) used for mixing the sulfuric acid, hydrogen peroxide and hydrogen fluoride solutions in each of the primary references such that the temperature rise of the resulting solution was 3 °C or less than the temperatures of the original components? We answer this question in the negative for the reasons discussed below.

⁷ With respect to each ground of rejection, Appellant specifically states that the applied prior art, alone or in combination, "does not teach, show, suggest, or make obvious mixing or combining an aqueous solution comprising sulfuric acid and hydrofluoric acid, wherein a concentration of the sulfuric acid in the aqueous solution is about 70% or less by weight, to form an intermediate solution at a predetermined temperature of about 3° C or less higher than the temperatures of the aqueous solution and the hydrogen peroxide solution." (Reply Br. 7, 8, 9, 10, 11, 12 (grounds of rejection 2-7); *see* Reply Br. 5-6 (ground of rejection 1).)

The Examiner found, and Appellant does not dispute, that it was well known in the art at the time of the invention that addition of water to a highly concentrated solution of sulfuric acid would produce an exothermic reaction. (Ans. 17; *see also* Spec. ¶¶ [0015] and [0018].)

Rath and Ramachandran disclose preparation of cleaning solutions “by admixing an aqueous sulfuric acid such as a 98 percent by weight solution with an aqueous solution of hydrogen peroxide such as a 30 percent by weight solution and aqueous hydrofluoric acid such as 49 percent by weight and adding these solutions to water in an amount to provide the desired percentage of the sulfuric acid, hydrogen peroxide, and hydrofluoric acid” (Rath ‘074, col. 4, ll. 9-15; Ramachandran, p. 6, ll. 5-9).

Kuhn-Kuhnenfeld discloses that “[t]he simplest manner to obtain [] etching solutions is by the mixing of aqueous hydrofluoric acid and aqueous H₂O₂ of commercially-obtainable concentrations, and subsequent slow stirring of concentrated aqueous sulfuric acid, that is of about 98% by weight, into the mixture” (col. 1, ll. 48-53).

Kuhn-Kuhnenfeld discloses a first method of preparing a cleaning solution by mixing diluted hydrofluoric acid and hydrogen peroxide, and then adding a required quantity of concentrated sulfuric acid (98% by weight) “within about 2 to 5 minutes, while stirring” (col. 2, ll. 3-8).

Kuhn-Kuhnenfeld notes that when this preparation method is utilized, the cleaning solution should always be prepared freshly because it is not stable for any length of time due to the temperature increase which occurs upon addition of the sulfuric acid (col. 2, ll. 13-17).

Kuhn-Kuhnenfeld also discloses a second preparation method which is described as advantageous in that the cleaning solution is stable for

several days (col. 2, ll. 21-24). In this alternative preparation method, 98% by weight aqueous sulfuric acid is “slowly poured” into a solution of hydrofluoric acid and hydrogen peroxide, such that the resulting solution contains “considerably less sulfuric acid” than the solution prepared by the above-described first method (col. 2, ll. 27-30). Prior to use, the cleaning solution must be heated (col. 2, ll. 29-32).

“[D]iscovery of an optimum value of a variable in a known process is usually obvious.” *Pfizer, Inc. v. Apotex, Inc.*, 480 F.3d 1348, 1368 (Fed. Cir. 2007); *see also, In re Geisler*, 116 F.3d 1465, 1470 (Fed.Cir.1997); *In re Boesch*, 617 F.2d 272, 276 (CCPA 1980). The rationale for determining the optimal parameters for prior art result effective variables “flows from the ‘normal desire of scientists or artisans to improve upon what is already generally known.’” *Id.* (quoting *In re Peterson*, 315 F.3d 1325, 1330 (Fed. Cir. 2003)). It is normally expected that a change in temperature, or concentration, or both, would be an unpatentable modification over the prior art. *In re Aller*, 220 F.2d 454, 456 (CCPA 1955). Therefore, when patentability is predicated upon a change in a condition of a prior art composition, such as a change in concentration or the like, the burden is on Appellant to establish with objective evidence that the change is critical, i.e. it leads to a new unexpected result. *See In re Woodruff*, 919 F.2d 1575, 1578 (Fed. Cir. 1990); *In re Aller*, 220 F.2d at 456.

Appellant argues that Ramachandran, Rath, and Kuhn-Kuhnenfeld describe methods of forming cleaning solutions by mixing concentrated sulfuric acid, 98% by weight, with other components including hydrogen peroxide. (App. Br. 12, 14, 16, and 18.) According to Appellant, mixing concentrated sulfuric acid with hydrogen peroxide and other components in

the concentrations disclosed in the references would produce an exothermic disassociation, resulting in an intermediate solution having a temperature which exceeds the recited limitation of claimed requirement of a “temperature of about 3°C or less higher than temperatures” (claims 1, 14, and 29) of the original components. (App. Br. 12, 14-15, 16, 18-19, and 20-21.)

Appellant’s argument is not persuasive because Appellant has not addressed the facts and reasons relied on by the Examiner in rejecting the claims. The Examiner’s obviousness determination is not based on a finding that the references explicitly disclose the claimed sulfuric acid concentration or a temperature increase of about 3°C or less upon mixing. Rather, the Examiner’s position is that it would have been within the level of skill of the ordinary artisan to have adjusted various mixing parameters in preparing the prior art cleaning solutions so as to minimize the effect of the resultant exothermic disassociation on temperature of the solution. In particular, the Examiner asserts that the ordinary artisan would have understood that the rise in temperature of the solution could be more readily controlled by first diluting the highly concentrated sulfuric acid solutions of the references and/or by a more gradual mixing of the sulfuric acid and hydrogen peroxide solutions. (Ans. 17.)

We view the Examiner’s position as reasonable, particularly given Kuhn-Kuhnenfeld’s disclosure that a more stable cleaning solution may be produced by slowing the rate of addition and using a lesser quantity of sulfuric acid. (*See supra*, pp. 6-7.) Appellant has not provided persuasive arguments or evidence to refute the Examiner’s finding that adjustment of process parameters, such as concentration and rate of addition, would have

been a matter of routine optimization in preparing the prior art cleaning solutions. Nor has Appellant attempted to show that such routine optimization would not have resulted in Appellant's claimed intermediate solution.

In view of the foregoing, we conclude that Appellant has not identified reversible error in the Examiner's obviousness determination as to independent claims 1, 14, and 29. Therefore, we sustain the rejections of claims 1, 2, 5, 9-12, 14, 20, and 24-27 under 35 U.S.C. § 103(a) as unpatentable over Ramachandran; claims 1, 2, 5, 9-12, 14, 20, and 24-27 under 35 U.S.C. § 103(a) as unpatentable over Rath '074; claims 1, 2, 5, 9-12, 14, 20, 24-27 and 38 under 35 U.S.C. § 103(a) as unpatentable over EP '081; claims 1, 2, 5, 9, 10, 14, 20, 24, 25, and 38 under 35 U.S.C. § 103(a) as unpatentable over Kuhn-Kuhnenfeld; and claims 3, 4, 17-19, 29, 30, 34, 35, 40, 42, 43, and 45 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran or Kuhn-Kuhnenfeld in view of Gotoh.

We have separately considered the rejections of dependent claims 13 and 28 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran in view of Oonishi and dependent claims 36 and 37 under 35 U.S.C. § 103(a) as unpatentable over Rath or Ramachandran in view of Gotoh as applied to claim 34, and further in view of Oonishi. However, because Appellant has not presented additional arguments in support of patentability of claims 13, 28, 36, and 37 (*see* App. Br. 22 and Reply Br. 10-12), we likewise sustain these grounds of rejection.

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The decision of the Examiner rejecting claims 1-5, 9-14, 17-20, 24-30, 34-38, 40, 42, 43, and 45 is affirmed.

No time period for taking any subsequent action in connection with this appeal may be extended under 37 C.F.R. § 1.136(a)(1)(iv).

AFFIRMED

PL Initial:
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